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THESIS

DETERMINATION OF THE CROSSLINKING G-FACTOR FOR
POLYSTYRENE USING ULTRA-RELATIVISTIC ELECTRONS

by

Armand Marcel Pelletier

June 1968

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DETERMINATION OF THE CROSSLINKING G-FACTOR FOR
POLYSTYRENE USING ULTRA-RELATIVISTIC ELECTRONS

by

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Submitted in partial fulfillment of the
requirements for the degree of

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June 1968

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ABSTRACT

The crosslinking G-factor for polystyrene using 80-90 MeV electrons was determined for comparison with the crosslinking G-factor determined using pile radiation with neutron energies up to 2 MeV, and Co^{60} gamma rays. The high energy electron source used was the N.P.G.S. linear accelerator.

A G-factor of $.035 \pm .006$ was determined. This value is slightly lower than the value of $.05 \pm .01$ reported for pile and gamma radiation (1).

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1. INTRODUCTION

Factors Affecting Crosslinking and Degradation in Polystyrene

When a material is subjected to ionizing radiation many different effects may be produced, depending partly on the type of radiation employed and partly on the material irradiated.

In polymers the principal events of interest are essentially chemical changes induced in the polymer chains by the ionizing radiation. These include: crosslinking or the formation of covalent bonds between different chains; degradation or the breaking of primary bonds along the polymer chain; and evolution of gases, primarily hydrogen, liberated from the polymer molecule.

Such chemical events are characterized primarily by the energy transferred from the incident ionizing particle. The events can be related to dose by:

$$N_i = G_i D$$

where N_i is the number of events of type i which occur after the material is exposed to a dose D (conventionally measured in terms of 100 eV deposited in this context) and G_i is the so-called G-factor for the event i .

The process of crosslinking by radiation involves the formation of covalent bonds between the polymer chains. This process continues until an infinite, three-dimensional network is formed when the network is no longer soluble but only swells in solvents which dissolved the unirradiated material. A certain number of crosslinks must be formed before a sufficiently large network is formed to give a gel and consequently, there is a minimum radiation dose that must be deposited before any gel can be detected. The point where gel just

begins to form is called the gel point and the dose at which this happens is called the gel dose.

As the crosslinks are distributed in random fashion, some molecules will have no crosslinks while a few molecules will have several and it is these that give rise to the gel. Once the gel point has been reached the proportion of insoluble material increases rapidly. The actual shape of the dose response curve depends upon the molecular weight distribution of the molecules in the polymer. When the molecular weight distribution is uniform, (i.e., all the molecules in the polymer have identical molecular weights) the gel formation is most rapid.

No satisfactory reaction scheme has yet been suggested which fully explains the phenomenon of crosslinking. The fact that crosslinking has been shown to be dose-rate independent (1) imposes serious limitations on the list of possible mechanisms. The simplest way of forming a crosslink is by the combination of two radicals, but the probability of two activated entities being produced independently and in sufficiently close proximity to form a link varies as the square of the dose. Also, as the lifetime of radicals is finite because they can be lost by parallel reactions, a crosslinking process by radical recombination would be dose-rate dependent. However, the difficulty of dose rate independence disappears if, as Charlesby postulates (1), the free radical wanders along the chain from carbon atom to carbon atom until it comes adjacent to another radical where a crosslink is formed. Electron spin resonance techniques have provided some evidence which seems to support this theory.

Another process associated with irradiation of polymers is degradation. Degradation is essentially the reverse of cross-linking and is characterized by random fracture of the polymer chain with a rearrangement of the atoms near the point of fracture to stabilize the end-groups. This process is essentially different from the process of depolymerization (often produced by thermal means) in which a change in one of the bonds allows the molecule to revert wholly or in part to the original monomer. In radiation induced degradation little or no monomer is produced even after extensive main chain fracture. Depolymerization is a chain reaction, involving many of the bonds present in a polymer molecule, whereas, degradation only affects atoms in the neighborhood of the fracture site, and is best shown by a progressive reduction in the average molecular weight.

Both crosslinking and degradation can occur simultaneously in a given substance, however, one process generally predominates, which leads to a broad categorization of polymers into two classes: those which degrade and those which crosslink. In polystyrene the principal result of ionizing radiation is the formation of cross-links. Some degradation, however, has been observed in a number of experiments where samples, having large surface area to volume ratios, were irradiated in air (2). However, this effect can usually be minimized by proper choice of sample geometry.

Several other factors which effect the process of crosslinking and which must be considered in the experimental design have been studied. Temperature effects on the degree of crosslink formation in polystyrene have been reported by Pravendnikov et. al. (3).

They noted an increase of about 50% in the degree of crosslinking when a sample of polystyrene irradiated at 25°^oC was heated above the glass temperature. This effect is assumed to be related to an increased mobility of free radicals which were trapped at lower temperatures. Also they found a drastic reduction in the efficiency of crosslinking for samples irradiated at 130° to 140°^oC. This effect has been interpreted on the assumption that at these high temperatures the radicals reacted primarily by disproportionation instead of combining. In the design of the experiment reported here such effects were obviated by choice of a sample geometry which insured a small temperature rise during irradiation.

The effect of dose rate on G-factor has been investigated by Charlesby (1). He concludes that the degree of crosslinking in polystyrene is independent of dose-rate over a 10⁵ range of radiation intensities.

Another variable that has been proposed as a possible parameter in crosslinking efficiency is the energy of the incident radiation. Essentially all previous determinations of crosslinking efficiency have been done with low energy ionizing radiation. The purpose of the investigation reported in this thesis was to determine the G-factor for crosslinking in polystyrene when the irradiating particles are electrons with energies of 80-90 MeV so as to compare with the G-factor determined with lower energy radiation. This investigation was prompted by previous work (4) which indicated that a different G-factor from that determined with lower energies might be obtained when the ionizations were produced by ultra-relativistic electrons.

The G-Factor

The method for obtaining the G-factor used in this investigation was the determination of gel point. Gel begins to form when, on the average, each polymer chain is connected by one crosslink so that the average molecular weight between crosslinks, M_c , is equal to the initial weight average molecular weight M_w .

The average distance between crosslinks, calculated by M_c , is easily related to the dose D expressed in megarads (a rad is a unit of energy deposited equal to 100 eV per gram of any material) by:

$$M_c = \frac{.48 \times 10^6}{G D} \quad (1)$$

The criterion then for first gel formation is that $M_c = M_w$. Thus if D_g is the dose corresponding to the first formation of gel, the G-factor will be given by:

$$G = \frac{.48 \times 10^6}{M_w D_g} \quad (2)$$

Other techniques exist for measuring the G-factor by the direct determination of M_c , primarily through the swelling technique. In this technique the polymer gel is swollen by soaking it in a "good solvent". A solvent is good if the solvent molecules have a greater attraction for the units of the polymer chain than do the chain units for each other so that in equilibrium, an amount of solvent will be imbibed which is related to the flexibility of the polymer chain (i.e., its ability to distort to allow the interaction of solvent molecules with the polymer chains). The amount of solvent imbibed is inversely related to M_c and directly related to the strength of the polymer-solvent interaction (5). For high accuracy

this technique requires a good knowledge of the polymer-solvent interaction parameters and so we did not employ the technique in this work. Further, for doses in the neighborhood of D_g , which was the area of primary concern here, the technique becomes quite limited by the available measuring techniques.

2. EXPERIMENTAL PROCEDURE

Design and Irradiation

A compromise in sample size had to be made. A sufficiently large sample to make accurate weight and gel determinations and a sample with fairly large volume to surface area ratio to minimize degradation was essential. However, a sample too large would greatly enhance the problem of temperature rise during irradiation. After preliminary heat transfer calculations, a cylindrical sample 1.3 cm. in diameter and 3 mm. thick was chosen. A thermocouple was imbedded in one such sample and it was irradiated to determine what the temperature rise would actually be. The temperature rise was approximately 10°C above ambient temperature of 21°C . This was well below the softening temperature of 80°C for polystyrene and so this sample size was used throughout the investigation.

The samples were irradiated by an electron beam produced by the N.P.G.S. linear accelerator. The samples were irradiated end-on, the impinging electron beam being centered on and directed along the axis of the cylindrical sample. A major problem was that the beam was not uniform over the sample. The horizontal and vertical profiles of the beam were measured using a remote-controlled apparatus which allowed two mutually perpendicular copper wires to be traversed across the center of the electron beam. The variation of the voltage developed between the wires and the grounded part of the apparatus as the wires traversed across the beam was recorded on a Honeywell Electronic 19 recorder. This variation was taken to be proportional to the electron density across the beam. The voltage variation in the

vertical wire gave the horizontal profile. The vertical profile was obtained from the variation in voltage in the horizontal wire.

The vertical and horizontal profiles showed that the electron density across the beam closely followed a gaussian distribution and that the contours of constant electron density were elliptical. Figure 1 shows the electron density distribution and its relation to the size of the samples.

Calculation of total current flux through the samples were made using values of total integrated current (measured by a Carry 401 vibrating reed electrometer connected to a secondary emission monitor (SEM) located in the beam) and the cross section of beam interrupted by the sample. Average current values (measured by a Beckman microampmeter connected to the SEM) varied between 2 and 3 micro-amps. The energy of the electrons (estimated from nuclear magnetic resonance probe measurements of the magnetic field strength of the magnets bending the beam) varied between 80 and 90 MeV from one irradiation to another.

Determination of Weight Average Molecular Weight

Weight average molecular weight (M_w) determinations for polystyrene were made using intrinsic viscosity techniques (6). Since the effect of the rate of shear on the observed specific viscosities should be small for polymers having intrinsic viscosities in the range of those found in this investigation, i.e., $\eta < 3$, correction to zero rate of shear was not applied. Intrinsic viscosity (limiting viscosity number, LVN) is defined by:

$$[\eta]_o = \lim_{c \rightarrow 0} \frac{\eta - \eta_o}{c} \quad (3)$$

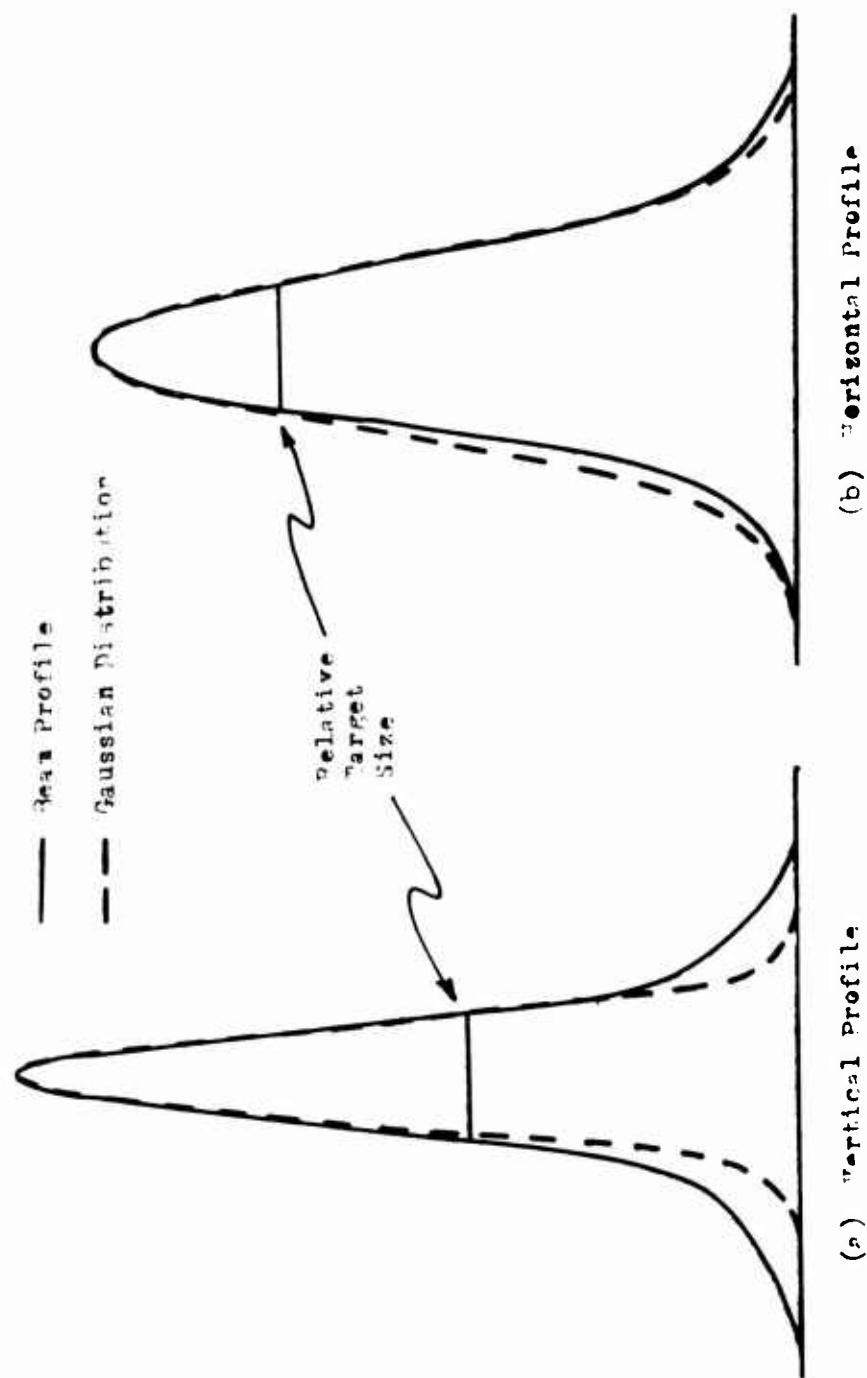


Fig. 1 Electron beam profile

where:

$[\eta]_0$ = intrinsic viscosity number (LVN)

η_0 = viscosity of solvent

η = viscosity of polymer solution

c = concentration of polymer solution in grams per 100 ml.

Because of its excellent properties as a solvent for polystyrene, and because the various parameters which were needed (see eq. 4) have been well established (5), benzene was chosen as the solvent for these determinations.

The empirical equation relating intrinsic viscosity to molecular weight M for a polymer of uniform molecular weight is (1):

$$[\eta]_0 = K M^a \quad (4)$$

where K and a are temperature dependent constants of the polymer-solvent system. For molecularly heterogeneous polymers, eq. 4 gives the viscosity average molecular weight M_{η} , where M_{η} is defined by:

$$M_{\eta} = \left[\frac{\sum n(M) M^{1+a}}{\sum n(M) M} \right]^{1/a} \quad (5)$$

where $n(M)$ is the number of molecules of weight M. The corresponding viscosity average degree of polymerization u_{η} is given by:

$$u_{\eta} = \frac{M_{\eta}}{w} = \left[\frac{\sum n(u) u^{1+a}}{\sum n(u) u} \right]^{1/a} \quad (6)$$

where: w = the molecular weight of a monomer unit

$n(u)$ = the number of molecules with u monomer units

$u = M/w$

For the general distribution:

$$n(u) = C \left(\frac{u}{u_1}\right)^{\lambda-1} \exp(-\lambda \frac{u}{u_1}) \quad (7)$$

where:

C = a normalizing factor

$$u_1 = M_n / w$$

where M_n = number average molecular weight

λ = a parameter defining the type of distribution

Summing eq. 6 using eq. 7 and

$$u_2 = \frac{M_w}{w} = \frac{\sum n(u) u^2}{\sum n(u) u} \quad (8)$$

it can be shown that:

$$M_w = \left[\frac{\Gamma(\lambda+1)}{\Gamma(\lambda+a+1)} \right]^{1/a} \left[\frac{\Gamma(\lambda+2)}{\Gamma(\lambda+1)} \right] M_\eta \quad (9)$$

where Γ is the gamma function. When λ tends to infinity, the distribution becomes uniform. For a random distribution (a molecular distribution obtained by random fracturing of an infinite chain or as a result of many polymerization reactions), $\lambda = 1$, while for a pseudo-random distribution $\lambda = 0$. Hence for a random distribution using $a = .74$:

$$M_w = 1.06 M_\eta \quad (10)$$

while for a pseudo-random distribution:

$$M_w = 1.23 M_\eta \quad (11)$$

Viscosity measurements of the polystyrene-benzene solutions were made using a Cannon-Fenske capillary viscometer. Viscosity is related to flow time by:

$$\frac{\eta}{\rho} = At + \frac{B}{t} \quad (12)$$

where

η = solution viscosity

ρ = solution density

t = flow time

The constants A and B are characteristic of the viscometer and were determined by calibration with water. Since the variation of ρ was negligible, the value of η/ρ calculated from eq. 12 was used directly in eq. 3. The constant B was determined to be approximately zero. As a result eq. 3 was used in the following form:

$$[\eta]_o = \lim_{c \rightarrow 0} \frac{t - t_o}{c t_o} \quad (13)$$

where

t = flow time for polystyrene-benzene solution

t_o = flow time for pure benzene

The value of $[\eta]_o$ was obtained graphically by plotting $(t - t_o)/ct_o$ as a function of c and taking the intercept at $c = 0$ as $[\eta]_o$ (see Fig. 2). Equation 4, (with $M = M_{\eta}$), eq. 10, and eq. 11 were then used to calculate M_w . M_w of the polystyrene used assuming a random distribution was determined to be $(1.73 \pm 0.1) \times 10^5$. Assuming a pseudo-random distribution, M_w was found to be $(2.00 \pm 0.1) \times 10^5$.

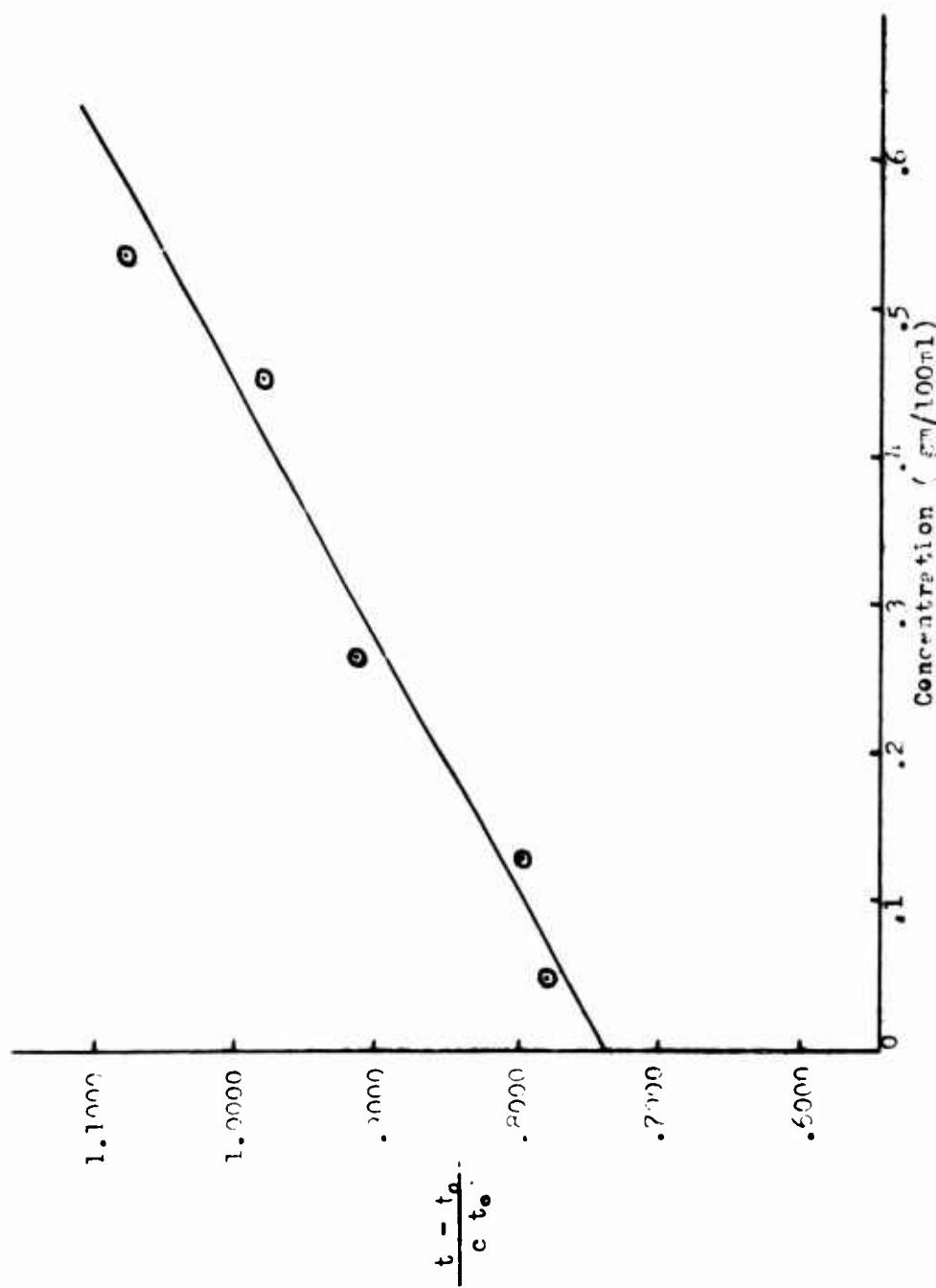


Fig. 2 Determination of Limiting Viscosity Number (LVN)

Theoretical Sol Fraction Dependence on Dose

Sol fraction, S , is that fraction of the original sample that dissolves in the solvent. Assuming a pseudo-random molecular weight distribution the sol fraction is given by (1):

$$S = 1 \text{ for } \delta < 1$$

$$S = 1/\delta \text{ for } \delta \geq 1$$

where $\delta = D/D_g$.

If we define D_o to be the dose at the center of the sample for which gel is just formed then the variation of δ over the sample is:

$$\delta(x,y) = (D/D_o) \exp \left[-\frac{x^2}{a^2} - \frac{y^2}{b^2} \right] \quad (14)$$

where a and b are determined from the beam profile. These parameters varied slightly between the first and second series of irradiations. Since the contours of equal dose were elliptical, of particular interest is the ellipse on which $\delta = 1$. This ellipse has semimajor axis α and semiminor axis β given by:

$$\alpha = a [\ln(D/D_o)]^{\frac{1}{2}} \quad (15)$$

$$\beta = b [\ln(D/D_o)]^{\frac{1}{2}} \quad (16)$$

The total sol fraction is given by:

$$S = [1/(\pi r_o^2)] \iint S(x,y) dA \quad (17)$$

This integral must be evaluated for three different cases:

Case 1: $0 \leq \alpha \leq r_o$ i.e. $D_o \leq D \leq D_1$

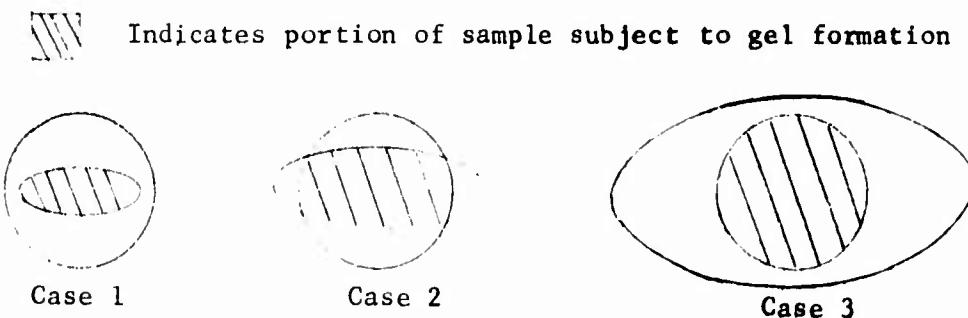
$$\text{where } D_1 = D_o \exp(r_o^2/a^2)$$

Case 2: $\beta < r_o < \alpha$ i.e. $D_1 \leq D < D_2$

$$\text{where } D_2 = D_o \exp(r_o^2/b^2)$$

Case 3: $r_o < \beta$ i.e. $D \geq D_2$

The physical significance of these three cases is illustrated in the following figures:



In case 1 the region of sample outside the ellipse is completely soluble. This contains an area:

$$A_1 = \pi r_o^2 - \pi \alpha \beta \quad (18)$$

The total sol fraction is then:

$$S = \frac{1}{\pi r_o^2} \left\{ \iint_{\text{inner ellipse}} (1/\delta) dA + \pi [r_o^2 - ab \ln(D/D_o)] \right\} \quad (19)$$

Let

$$I = \iint (1/\delta) dA = D_o/D \iint \exp [x^2/a^2 + y^2/b^2] dA \quad (20)$$

this integral is constant over the concentric ellipses

$$(x^2/a^2) + (y^2/b^2) = \xi^2$$

where ξ ranges from 0 to ξ_{\max} which is defined by:

$$\alpha^2/(\xi_{\max}^2 a^2) = 1 = \ln(D/D_o)/\xi_{\max}^2$$

On the ellipse where $0 \leq \xi_1 < \xi_{\max} = [\ln(D/D_o)]^{\frac{1}{2}}$, the integrand is given by $\exp(\xi^2)$. The element of area dA can be obtained by considering the area between the concentric ellipses of radius ξ and $\xi + d\xi$:

$$dA = 2\pi ab\xi d\xi \quad (21)$$

Substituting eq. 21 into eq. 20 and integrating we obtain:

$$I = (D_o/D) \pi ab [\exp(\xi_{\max}^2) - 1] \quad (22)$$

or:

$$I = \pi ab [1 - D_o/D] \quad (23)$$

Thus the sol fraction is:

$$S = 1 - (ab/r_o^2) [(D_o/D) + \ln(D_o/d) - 1] \quad (24)$$

The sol fraction for case 2 is difficult to determine directly, however, it is easy to determine an upper and a lower limit. The average of these values can then be taken as the sol fraction. This method yielded values which were in excellent agreement with the values calculated exactly for regions where case 2 overlapped case 1 and case 3.

For the lower limit (S_{LL}) we assume $\delta = (D/D_o) \exp(-r^2/a^2)$ and therefore:

$$S_{LL} = (a^2/r_o^2) (D/D_o) [\exp(r_o^2/a^2) - 1] \quad (25)$$

The upper limit of sol fraction will result if we assume $\delta = (D/D_o) \exp(-r^2/b^2)$. Hence S_{UL} will be:

$$S_{UL} = 1 - (b^2/r_o^2) [\ln(D/D_o) + (D_o/D) - 1] \quad (26)$$

Therefore where the conditions for case 2 applied, the sol fraction was calculated using:

$$S = (S_{UL} + S_{LL})/2 \quad (27)$$

In case 3 the entire sample is irradiated beyond the gel point so we have:

$$S = (1/\pi r_0^2) \iint (D_0/D) \exp [-(x^2/a^2) - (y^2/b^2)] dx dy \quad (28)$$

Eq. 28 can be simplified to yield:

$$S = (4ab/r_0^2) (D_0/D) \int_0^{r_0/a} \exp(-q^2) dq G \left\{ (a/b) [(r_0^2/a^2) - q^2]^{1/2} \right\} \quad (29)$$

where:

$$G(z) = \int_0^z \exp(-t^2) dt$$

and $q = x/a$

Equation 29 was evaluated using numerical integration.

For a random distribution $S = 1$ for $\delta < 1$ and $S + \sqrt{S} = 2/6$ for $\delta \geq 1$. If we define $S + \sqrt{S} = S_{random}$ then:

$$S_{random} = 2S_{pseudo-random}$$

so that the above calculation can also be used for the case of the random distribution.

Experimental Determination of Sol Fraction

After irradiation the samples, which weighed approximately .2 grams each, were placed in a small tubular metal container into which small drain holes had been drilled. These containers were then placed into small glass beakers which contained 20 ml of benzene. The samples were kept in the benzene for three days. The metal

containers holding the gel were then removed, allowed to drain, and then quick frozen with liquid nitrogen and put under vacuum for six days to dry. The first three days the vacuum system was packed in ice and kept at 0^oC. During the last three days the temperature of the vacuum system was raised to 65^oC for final drying. The dried gel samples, which had a white porous appearance, were then weighed. The difference between the original sample weight and the dried gel weight divided by the original sample weight was taken as the sol fraction.

3. RESULTS AND CONCLUSION

Determination of Gelling Dose and G-Factor

The gelling dose was determined by comparison of measured sol fraction as a function of dose delivered to the center of the sample with the theoretical sol fraction vs dose curve for various assumed gelling doses. This was done assuming a random distribution and a pseudo-random molecular weight distribution (Figures 3-6). Statistical calculations (rms deviation) showed that the experimental data fits equally well the 70 Mrad-pseudo-random and the 80 Mrad-random curve. Taking into account all possible errors, the G-factor calculated from eq. 2, using 80 Mrad and M_w calculated from eq. 10, was $.035 \pm .006$. The G-factor calculated from eq. 2, using 70 Mrad and M_w calculated from eq. 11 was $.034 \pm .006$. Hence within experimental error these values are identical.

Conclusion

The value for the G-factor obtained here is slightly lower than the value reported by Charlesby of $.05 \pm .01$ for low energy (1-2 MeV) radiation (1). This lower value is not a result of irradiation temperature difference since, according to Pravednikov (3), the rate of crosslinking of polymer chains at temperatures below their softening temperatures is independent of temperature; in other words, the "thermal" H atoms do not participate significantly in the reactions leading to the formation of crosslinks at temperatures below the softening temperature.

Also the lower value cannot be attributed to oxygen effects since Alexander (2) demonstrated that oxygen effects in polystyrene becomes significant only when irradiations are performed on very thin

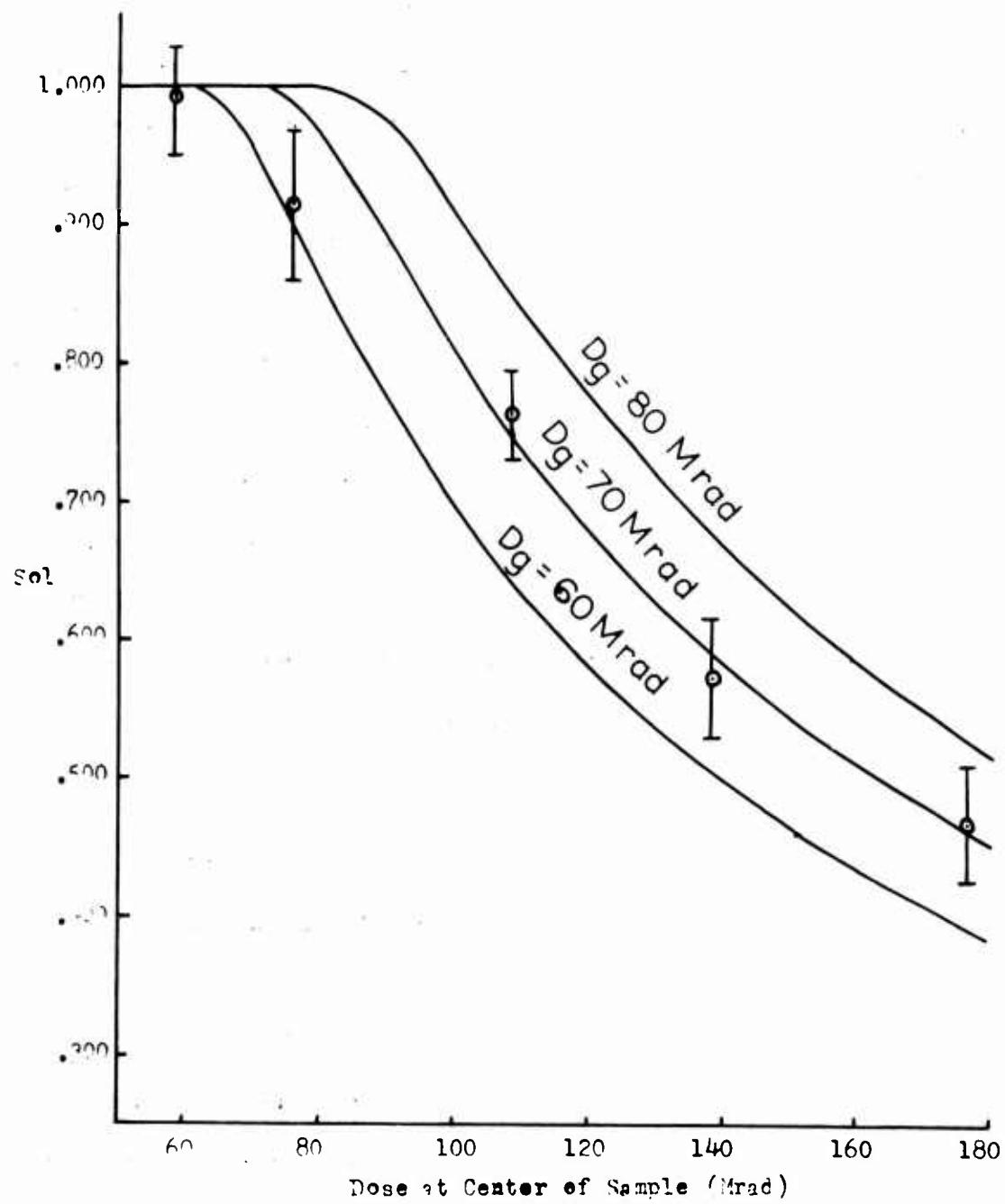


Fig. 3 Comparison of Pseudo-Random-Theoretical Sel vs Dose Curves with Experimental Data for Three Gel Doses

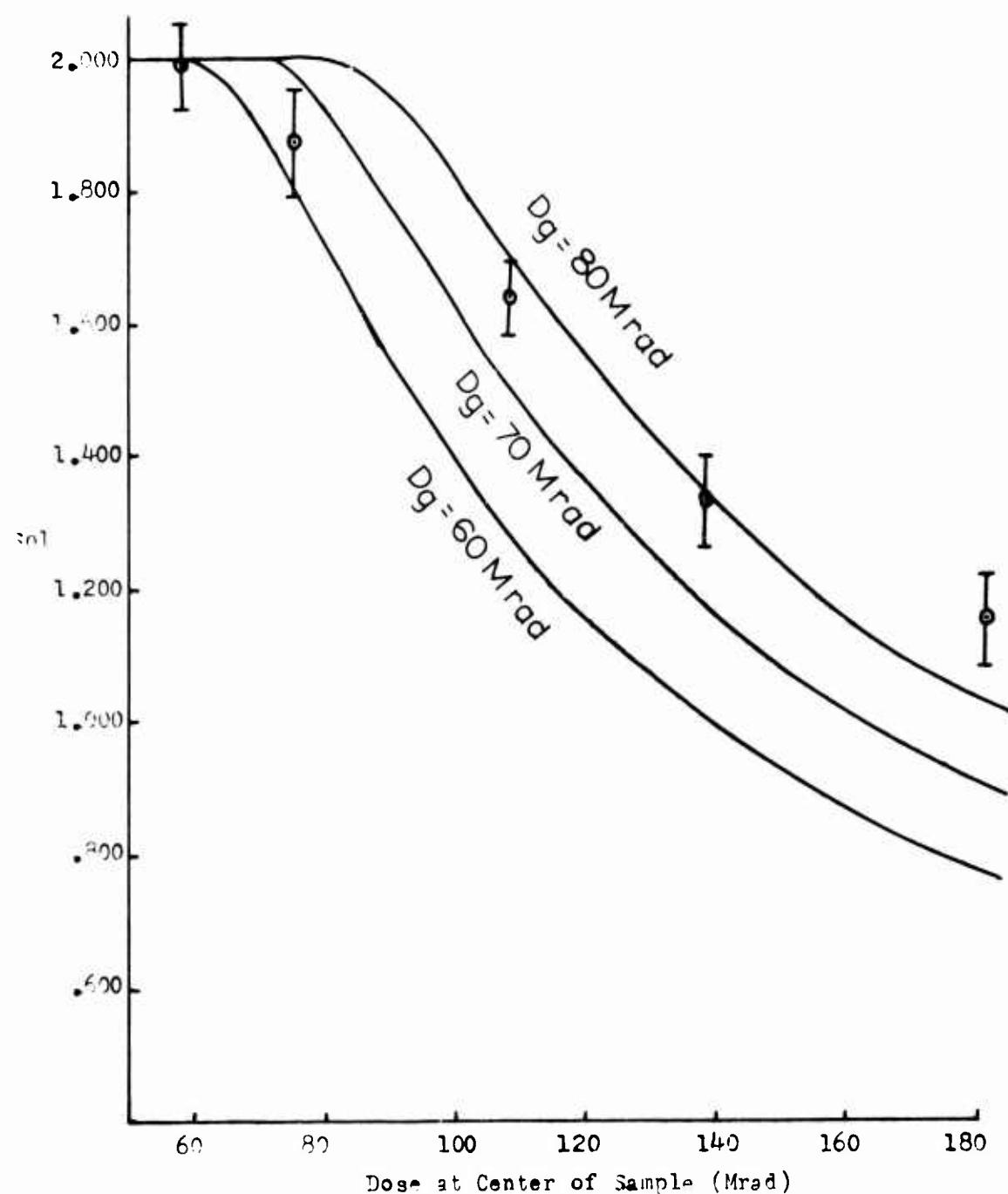


Fig. 4 Comparison of Random-Theoretical S_{01} vs Dose Curves with Experimental Data (S+VS) for Three Gel Doses

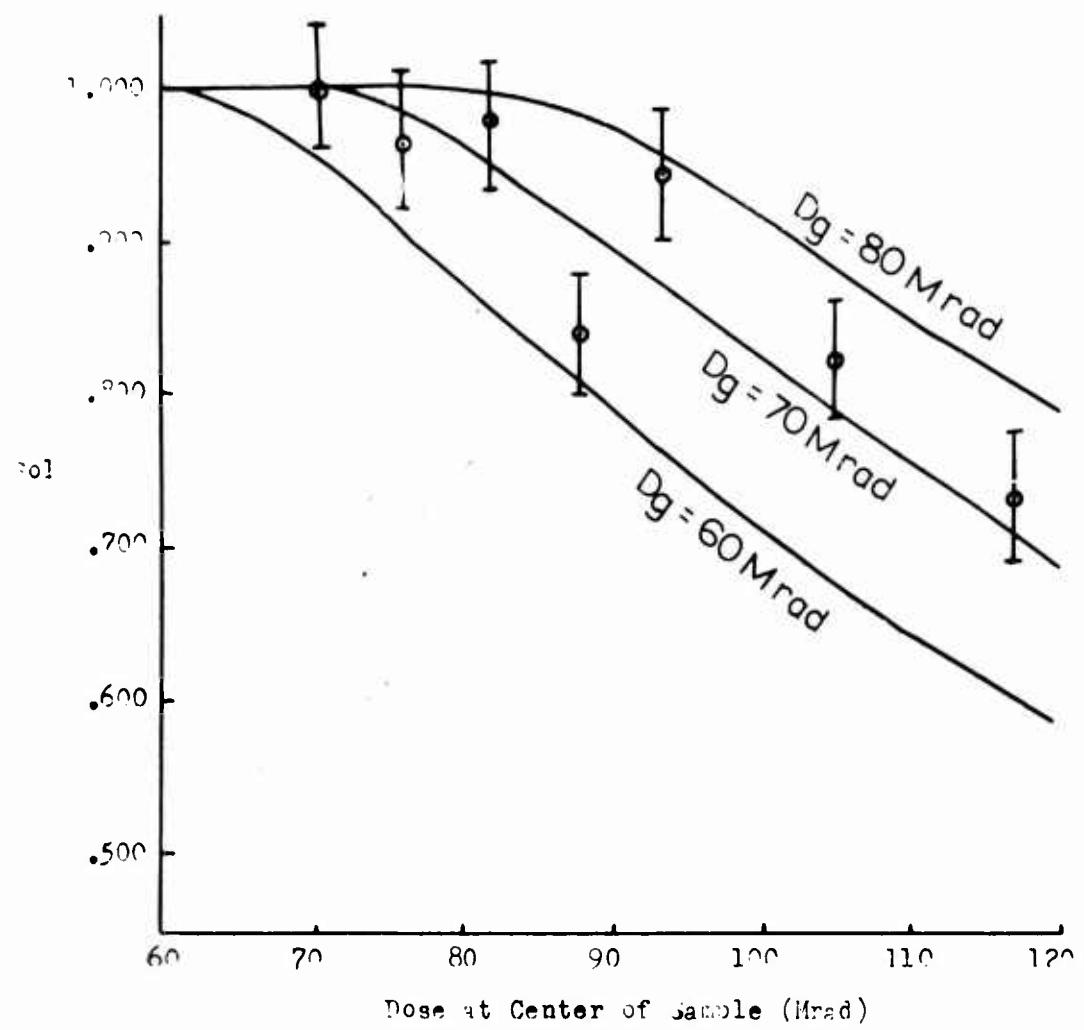


Fig. 5 Comparison of Pseudo-Random-Theoretical Sol vs Dose Curves with Experimental Data for Three Gel Doses

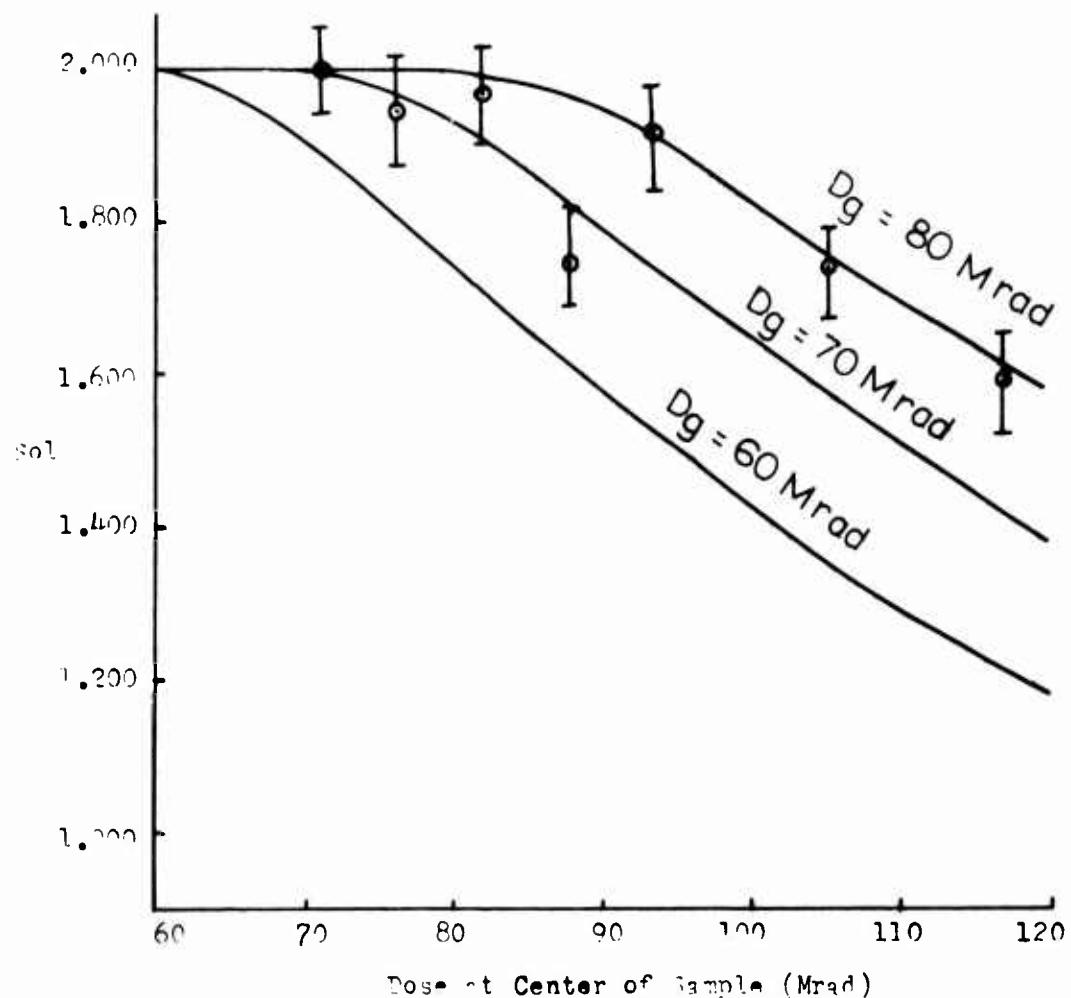


Fig. 6 Comparison of Random-Theoretical Sol vs Dose Curves with Experimental Data ($S+\sqrt{S}$) for Three Gel Doses

samples (approx. 200 microns thick). Hence it is concluded that the G-factor for crosslinking in polystyrene using ultra-relativistic electrons is slightly lower than the same G-factor determined using radiation with energy of the order of a few MeV.

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